

|  |                                      |               |
|--|--------------------------------------|---------------|
| 98-181205/17   | A60 D21 E31 G01 L02<br>(E11 G02 L01) | BADI 96.09.30 |
| BASF AG  |                                      |               |
| 96.09.30 96DE-1040188 (98.04.01) C09C 1/00, A61K 7/00, C03C 4/02, C09C 3/12, C09D 11/00, 7/12, C04B 33/14, C08K 9/06   | *EP 832943-A2                        |               |
| Blue tinted lustre pigments - are based on titanium di:oxide coated silicatic platelets heated in reducing atmosphere and reacted with alkoxy:silane compound (Ger)  |                                      |               |
| C98-058229 R(AL AT BE CH DE DK ES FI FR GB GR IE IT LI LT LU LV MC NL PT RO SE SI)   |                                      |               |
| Addnl. Data: KALIBA C, KELLER H, GONZALEZ GOMEZ J A, BIDLINGMAIER H, ELLINGHOVEN R, SCHMID R 97.09.24 97EP-116595  |                                      |               |
| Condensed moisture-resistant blue-tinted lustre pigments are based on titanium dioxide coated silicate platelets which have been heated in a reducing atmosphere and the reduced platelets then reacted with a silane of formula $R_nSiX_6$ (I) in which:<br>R = 1-10 C alkyl substituted in the $\omega$ -position by a glycidyl group, an amino group, a hydroxyl group or a monoalkylamino group or an alkoxy group in each of which the alkyl chain may contain up to 10C and may be interrupted by 1-5 ether O atoms or NH groups, and if a > 1 then the groups R may be the same or different; |                                      |               |

|   |              |
|---|--------------|
| A(8-E2) D(8-B) E(31-P2D, 31-P5, 35-K2) G(1-A6, 1-A8, 2-A3D, 2-A4A) L(2-G4)  |              |
| X = 1-4 C alkoxy;<br>a = 1 or 2;<br>b = 2 or 3; and<br>a + b = 4.   |              |
| <u>USE</u><br>The pigments are useful as colourants in paints, printing inks, inks, plastics, glass, ceramic products and decorative cosmetic products, especially colourants in automobile finishing lacquers. |              |
| <u>ADVANTAGE</u><br>The pigments have a high resistance to condensed moisture and good dispersibility in lacquers.  |              |
| <u>PREFERRED MATERIALS</u><br>Preferably in (I):<br>R = 3-glycidioxypropyl or 3-aminopropyl group;<br>X = ethoxy or methoxy;<br>a = 1; and  | EP 832943-A+ |

b = 3.

The silicate platelets are preferably of clear or white mica, and are especially of wet milled muscovite. The platelets may also be based on phlogopite, biotite, synthetic mica, talcum or glass platelets. The platelets have a mean largest diameter of about 1-200, preferably 5-100  $\mu\text{m}$ , a thickness of 0.1-1, preferably 0.5  $\mu\text{m}$  and a BET surface of 1-15, preferably 3-12  $\text{m}^2/\text{g}$ .

The thickness of the  $\text{TiO}_2$  coating layer is 50-100  $\mu\text{m}$  (silver) or 300-400  $\mu\text{m}$  (blue). The  $\text{TiO}_2$ -coated platelets are reduced e.g. by heating at 750-850°C in ammonia gas or at >800 to 900°C in an ammonia/hydrocarbon gas mixture.

The reduction process causes reduction of the  $\text{TiO}_2$  to  $\text{Ti}_3\text{O}_5$ ,  $\text{Ti}_2\text{O}_3$ ,  $\text{TiO}$ ,  $\text{Ti}$  oxynitride and  $\text{Ti}$  nitride which due to their blue absorption colours together with the blue reflecting substrate platelets gives intensive blue tinted lustre pigments. Suitable reduced  $\text{TiO}_2$ -coated mica pigments are available commercially as "Paliocrom" (RTM; BASF).

The reduced coated platelets are preferably reacted with the silane (I) in the presence of water or water vapour. Preferably the platelets are reacted with the vapourised silane. Reaction with the vapourised silane may be carried out e.g. in a whirling bed reactor or in a mixer for solids equipped with a means for deagglomeration.

#### EXAMPLE

4 kg of a silver-reflecting  $\text{TiO}_2$ -coated mica pigment, reduced with ammonia gas at 800°C, was charged to a 50 l "Lodge" (RTM) mixer. An  $\text{N}_2$  stream of 300 l/hour was passed in via a silane evaporator containing 3-aminopropyltriethoxysilane at 170°C and a second  $\text{N}_2$  stream of 160 l/hour was passed in via a water evaporator at 85°C. After 65 minutes 218 g silane and 52 g water had been introduced. Silane vaporisation was then terminated while water vapour was passed in for a further 80 minutes until a total of 117 g water had been added. The resulting pigment had a hydrocarbon content of 0.8 wt.%. When the product was sieved < 50  $\mu\text{m}$  the coarse fraction amounted to 2 wt.%. Lacquers pigmented with the resulting pigment showed very good retention of colour and lustre in the Cleveland Humidity Test and upon immersion in water at 80°C for 24 hours. (JT)  
(7pp0513DwgNo.0/0)

EP 832943-A